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Mazdoor Kisan Shakti Sangathan

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“पुराने को छोड़ नये के तरफ”

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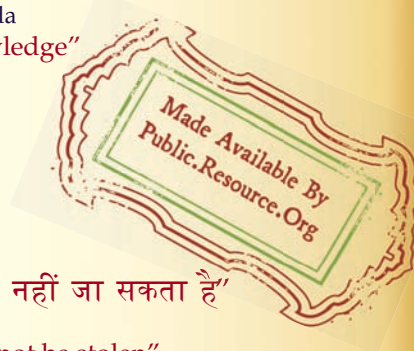
IS 15133 (2002): 2,6-Dichloro-4-Nitroaniline [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

2,6-डाईक्लोरो-4-नाइट्रोएनीलिन — विशिष्टि

Indian Standard

2,6-DICHLORO-4-NITROANILINE — SPECIFICATION

ICS 71.080.20

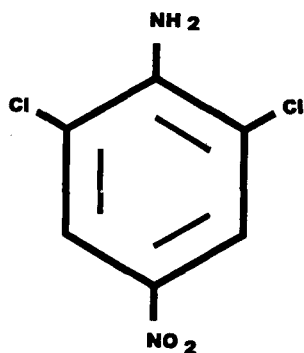
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NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dyes Intermediate Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

2,6-Dichloro-4-nitroaniline is an important dyestuff intermediate used in the manufacture of azo disperse dyes. It has the following structural formula:



2,6-DICHLORO-4-NITROANILINE

(MOLECULAR MASS: 207)

[CAS No. 99-30-9]

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

2,6-DICHLORO-4-NITROANILINE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for 2,6-dichloro-4-nitroaniline.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water (<i>third revision</i>)
5299 : 2002	Methods for sampling and tests for dye-intermediates (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of dry dull yellow powder.

3.2 The material shall also comply with the requirements given in Table 1.

**Table 1 Requirements for
2, 6-Dichloro-4-Nitroaniline**

Sl No.	Characteristics	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Assay by GC, percent by mass, <i>Min</i>	97.0	Annex A
ii)	2-Chloro-4-nitroaniline content by GC, percent by mass (on dry basis), <i>Max</i>	1.5	Annex A
iii)	4-Nitroaniline content, percent by mass (on dry basis), <i>Max</i>	0.5	Annex B
iv)	Matter insoluble in methanol, percent by mass, <i>Max</i>	0.2	11 of IS 5299
v)	Moisture content, % by mass, <i>Max</i>	0.5	10 of IS 5299

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material;

- b) Indication of the source of manufacture;
- c) Batch number;
- d) Gross, net and tare mass;
- e) Month and year of manufacture; and
- f) Cautionary note.

4.2.2 BIS Certification Marking

Each container may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in 4 of IS 5299.

5.2 Number of Tests

5.2.1 Tests for requirements of description and assay shall be conducted on each of the individual samples separately.

5.2.2 Tests for the determination of the remaining characteristics, namely, 2-chloro-4-nitroaniline, 4-nitroaniline, matter insoluble in methanol and moisture contents shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 For Individual Samples

The lot shall be declared as conforming to the requirements of the description and assay, if each of the individual test results (*see 5.2.1*) satisfies the relevant requirements given at 3.1 and in Table 1.

5.3.2 For Composite Samples

For declaring the conformity of the lot to the requirements of all other characteristics (*see 5.2.2*), while tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirement as given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see IS 1070*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, Sl No. (i) and (ii)]

METHODS OF TEST FOR 2, 6-DICHLORO-4-NITROANILINE, 2-CHLORO-4-NITROANILINE CONTENTS BY GAS LIQUID CHROMATOGRAPHY

A-1 OUTLINE OF THE METHOD

The assay of 2,6-dichloro-4-nitroaniline is determined by gas liquid chromatographic method. For detailed test procedure, reference may be made to IS 5299. However, the conditions for a typical chromatographic analysis are given below for guidance only.

A-1.1 Apparatus

Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram obtained with Perkin Elmer 8500 is attached.

The conditions are as follows:

- Column

:

Material — Stainless steel
Dimensions — Length: 2 000 mm, internal
Diameter — 1/8th inch
Stationary phase — 10 percent
UV 101 on chromosorb WHP,
mesh 80/100
- Carrier gas

:

Nitrogen
- Flow rate

:

30 ml/minute
- Injector temp.

:

300°C
- Detector temp.

:

300°C
- Condition

:

Oven temperature:
a) Initial temperature 150°C for
4 min, and
b) Final temperature 250°C
with a programming rate of
10°C/min
- Detector

:

Flame ionization
- Chart speed

:

5 mm/min
- Sample size

:

0.8 ml of 10 percent solution in
dimethylformamide
- Retention time

:

2-Chloro-4-nitroaniline — 9.94
min,
2,6- dichloro-4-
nitroaniline — 10.81 min
Unknown — 8.97 min

A-1.2 Interpretation of Chromatogram

The peak for 2-chloro-4-nitroaniline appears at 9.94 minute and for 2,6-dichloro-4-nitroaniline, it appears at 10.81 minute. A typical chromatogram under the conditions given above is shown in Fig. 1.

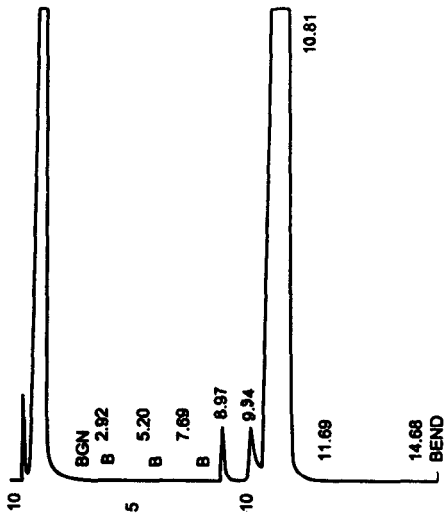


FIG. 1 TYPICAL CHROMATIC OF 2,6-DICHLORO-4-NITROANILINE

A-1.3 Calculation

Calculate the peak areas of individual constituent pertaining to 2,6-dichloro-4-nitroaniline as also the other constituents and calculate the purity/impurity of the sample as given below:

Assay, percent = $\frac{A}{A_1 + A_2 + A_3 + \dots + A_n}$

where

- A_1

=

Area under 2,6-dichloro-4-nitroaniline;
- A_2

=

Area under other peaks, say peak 2;
- A_3

=

Area under peak 3, etc; and
- n

=

number of other peaks observed apart
from 2,6-dichloro-4-nitroaniline.

A-1.3.1 Calculate the percentages of other constituents such as 2-chloro-4-nitroaniline in the similar manner.

ANNEX B

[Table 1, Sl No. (iii)]

DETERMINATION OF 4-NITROANILINE CONTENT BY
THIN LAYER CHROMATOGRAPHY METHOD**B-1 GENERAL**

4-Nitroaniline from 2,6-dichloro-4-nitroaniline is determined by thin layer chromatography method. For details of procedure for TLC, reference may be made to IS 5299.

B-2 REAGENTS

B-2.1 4-Nitroaniline — Prepare 0.01 percent solution (pure, reference) in acetone.

B-2.2 Eluent — Toluene — Ethyl acetate (96:4).

B-2.3 Nitrous Fumes Chamber — A glass chamber of size $250 \times 250 \times 250$ mm containing approximately 50 ml of concentrated HCl (30 percent *m/v*). The nitrous fumes are generated by adding a little solid sodium nitrite every time before inserting the plate.

B-2.4 Spray Reagent — 0.1 percent (*m/v*) solution of American base (N-1-naphthylethylene diamine dihydrochloride) in methanol.

B-3 APPARATUS

B-3.1 Thin Layer Chromatographic Chamber — $250 \times 100 \times 250$ mm.

B-3.2 Micropipette, of 10 ml capacity.

B-3.3 Pre-coated Thin Layer Chromatography Plates — TLC plates (200×200 mm) coated with silica gel about 0.25 mm thickness.

B-4 PROCEDURE

Weigh accurately 0.2 g of the sample (100 percent basic) and dissolve in 100 ml acetone. Take a plate coated with silica gel and spot 5 ml of each of the test solution and reference solutions along the start line at a distance of 3 cm from one another. Put the eluent in the chromatographic chamber. Insert the plate in the chamber and allow to run for about 30-35 minutes during which time the solvent front travels approximately 15 cm past the start line. Remove the plate and dry it in air. After drying put the plate in the nitrous fumes chamber for about 30 seconds. Remove the excess nitrous fumes by blowing air on it. Spray the plate with American base when the spot characteristics of 4-nitroaniline become visible at 0.23.

B-5 REPORTING

Estimate the quantity of the impurity by comparing the colour intensity of the spots of the material under test with that of the reference sample.

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Dyes Intermediate Sectional Committee, PCD 11

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The Gujarat Small Scale Dyestuffs Manufacturer's Association, Ahmedabad	SHRI R. S. PATEL SHRI R. R. SHAH (<i>Alternate</i>)
BIS Directorate General	SHRI ANJAN KAR, Director & Head (PCD) [Representing Director General (<i>Ex-officio</i>)]

Member-Secretary
DR (SHRIMATI) VIJAY MALIK
Director (PCD), BIS

Bureau of Indian Standards

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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